EFFECTS OF COLD-WORK ON THE MAGNETIC PROPERTIES OF NICKEL-ZINC FERRITE

Fifth Technical Report

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13. ABSTRACT

Cold-work is shown to have marked effects upon the magnetic properties of nickel-zinc ferrite. The effects can be rationalized in terms of residual elastic stresses together with the appearance of a stress-induced phase change.

Polishing appears to have a more severe effect than grinding, which may reflect a difference in the deformation characteristics of the two processes.

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I. INTRODUCTION

It has long been known that cold-work can exert a marked effect on the magnetic properties of metals, due partly to the introduction of crystal defects, but even more to residual elastic stresses. More recently, attention has been paid to the effects of cold work on the properties of ceramic magnetic materials such as ferrite, where it has been shown that grinding and polishing may alter the permeability[1] or coercive force[2] of these very brittle materials.

The problem first came to our notice during work aimed at producing nickel-zinc ferrite material for use in magnetic recording heads. Our initial characterization routines involved measurements of saturation magnetization, remanence, coercivity, and high-frequency permeability of toroid samples cut from ~0.025 inch-thick sheets of $Ni_{.36}Zn_{.64}Fe_2O_4$ sintered material. It was found that the results were significantly different from those measured by the head fabricators who employed much thinner slices, 0.007 inch, of the same material.

It was immediately suspected that the differences could be explained in terms of the relatively more severe cold-work experienced by the thinner samples. Consequently, annealing experiments were performed to see if the magnetic properties could be recovered. From Figure 1, which plots magnetic property values as a function of repeated cycles of polishing and annealing, it is apparent that magnetic changes are reversible in this material.

An interesting feature of these results is that all the magnetic properties appear to be structure-sensitive. It is understandable that coercivity, remanence, and low-frequency permeabilities should be affected by stress, since domain-wall mobility is known to be subject to magnetostrictive interactions; but saturation magnetization is usually considered a scalar quantity, virtually independent of crystalline defects such as dislocations or elastic strains.

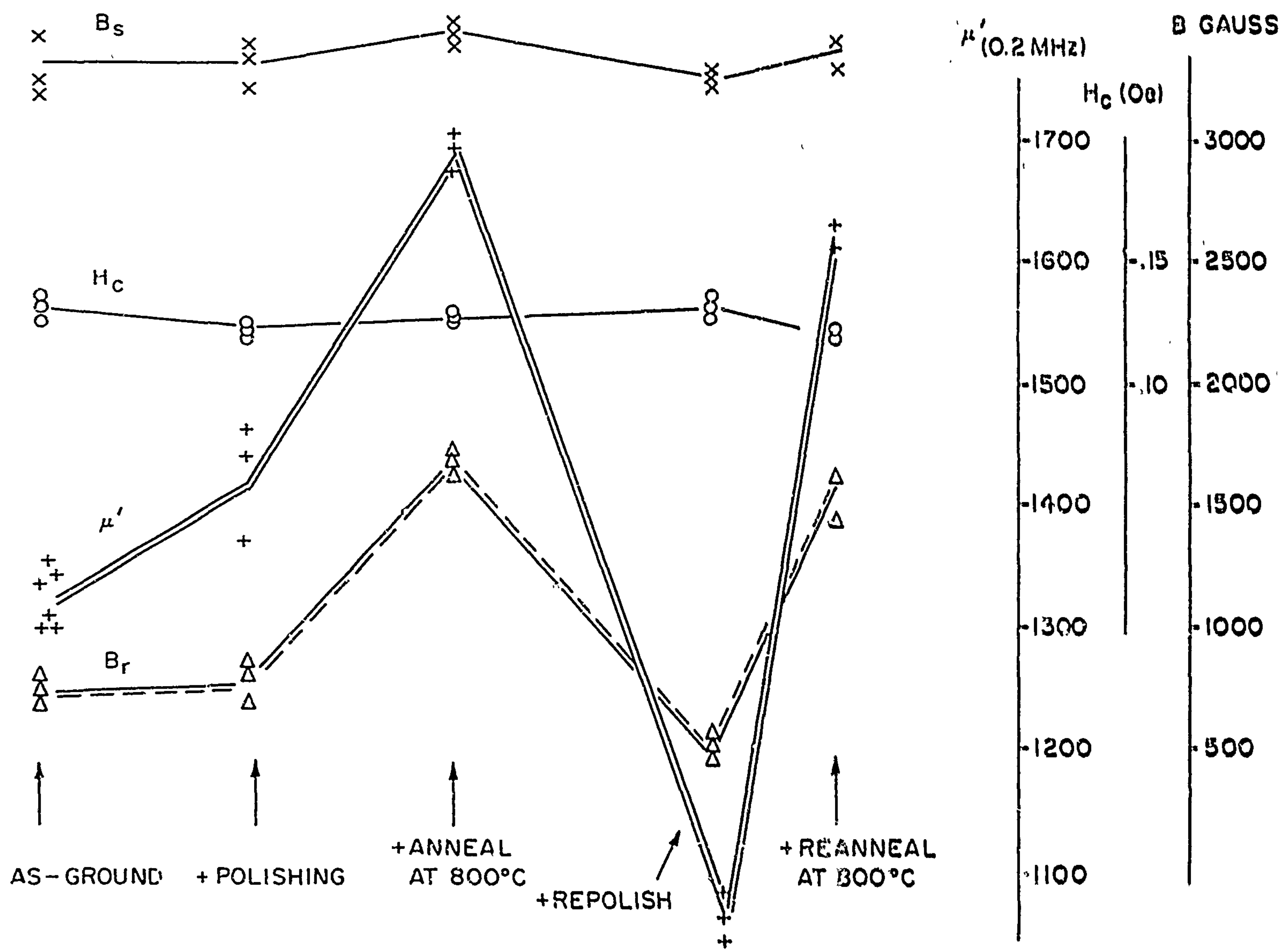

Figure 1. Recovery of Magnetic Properties by Annealing

In view of the technological importance of maintaining good reproducible magnetic characteristics of recording heads, an investigation of the mechanisms causing property changes was begun, along with an investigation of the kinetics of the annealing process.

It is the purpose of this report to discuss our results concerning the relationship between cold-work and magnetic property degradation; annealing effects will be considered in a later report.

II. EXPERIMENTAL METHODS

A. SAMPLE PREPARATION

Toroidal samples were prepared from sintered blocks of $Ni_{.36}Zn_{.64}Fe_2O_4$ composition by cutting thin slices from the blocks and then ultra sonic machining the toroids. Sample dimensions were fixed at 0.44 inch O.D. x 0.18 inch I.D. while the thickness was varied in the range from 0.050 inch down to 0.004 inch. Two techniques were used to prepare the toroid surfaces:

- Grinding on 600 grade silicon carbide paper
- Polishing with 10μ diamond-dust on a teak lap

B. MAGNETIC MEASUREMENTS

Saturation magnetization (B_S), remanence (B_R), and coercive force (H_C) were measured from 60 cycle hysteresis loops displayed on a cathoderay tube. The technique was quite standard, involving the winding of drive and pickup-coils on the toroids and the display of magnetic induction as a function of applied field.

High-frequency permeability measurements were made using the National Bureau of Standards resonant-cavity permeammeter[3], the bare sample serving to alter the resonance characteristics of the system. Frequencies of 0.2, 1, 1.25, 3, 5, 7, 10 and 20 MHz were employed, using special coils for each condition. The apparatus and method of calculating permeability are set out in reference (3).

C. STRUCTURAL INVESTIGATIONS

At a later stage in the investigation, transmission electron microscopy was used to examine cold-worked samples; thinning techniques based on those

described by Adda [4] were then developed. Briefly, these employed perchloric acid/acetic acid electrolytes and low applied voltages to prepare thin ferrite films by electro-polishing.

III. RESULTS

Raw data from the hysteresis loop measurements are shown in Figure 2, where B_S, B_r, and H_C are plotted as a function of sample thickness. Two effects are immediately clear:

- Magnetic properties vary with sample thickness in non-linear fashions
- Property changes are greater in polished, than in ground, samples at constant sample thickness.

In Figure 3 are plotted permeability measurements as a function of frequency for samples of different thickness. It may be noted from the diagram that permeability measurements are lower in polished samples than in ground ones, except at high frequencies (> 5 MHz).

Results on ground samples are replotted in Figure 4 as a function of sample thickness from which it will be clear that permeability changes at lower frequencies are non-linear with sample thickness.

Electron microscopy results will be described in a later section of this report, when their significance may be clearer.

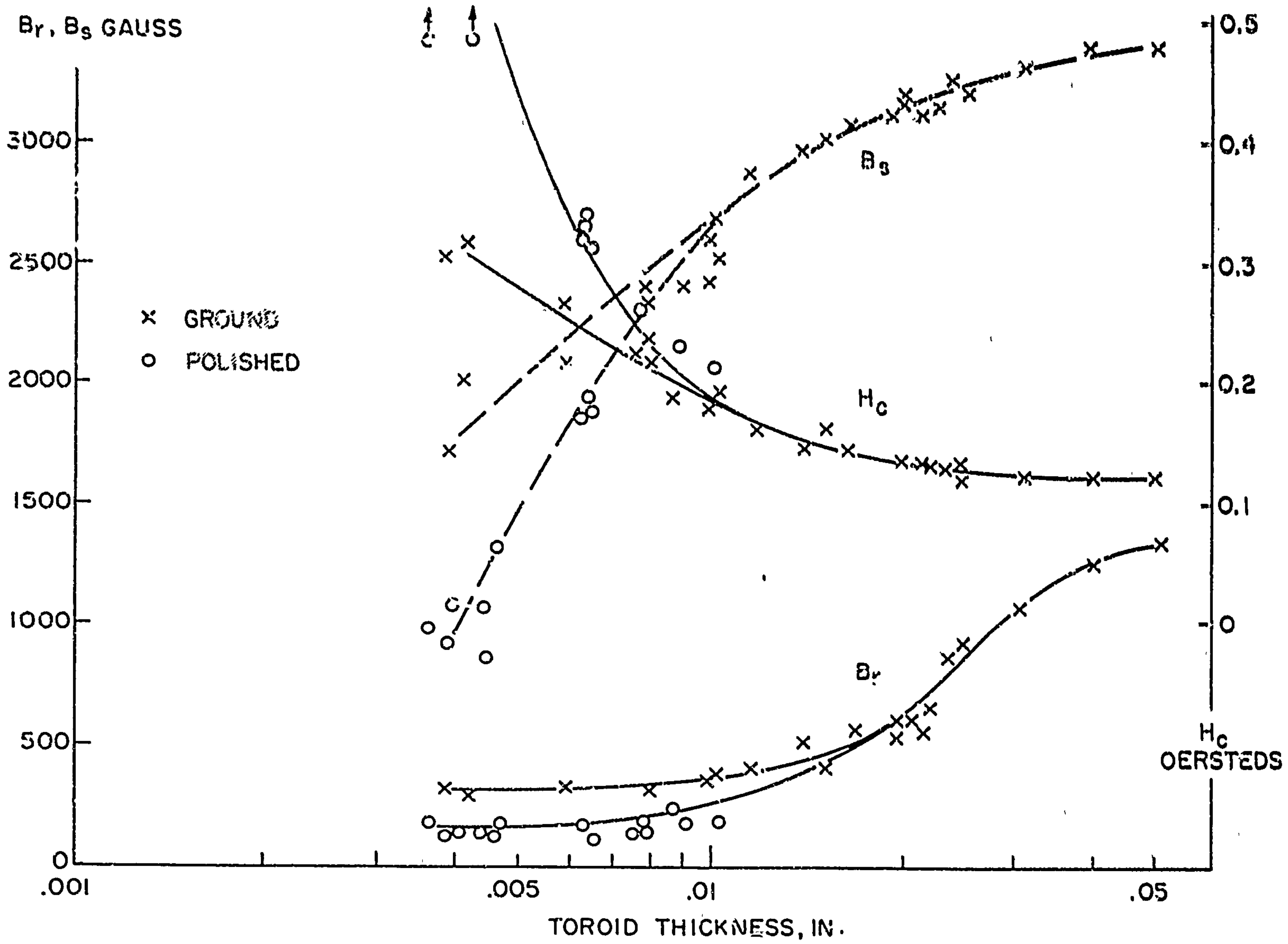

Figure 2. Magnetic Properties as a Function of Sample Thickness

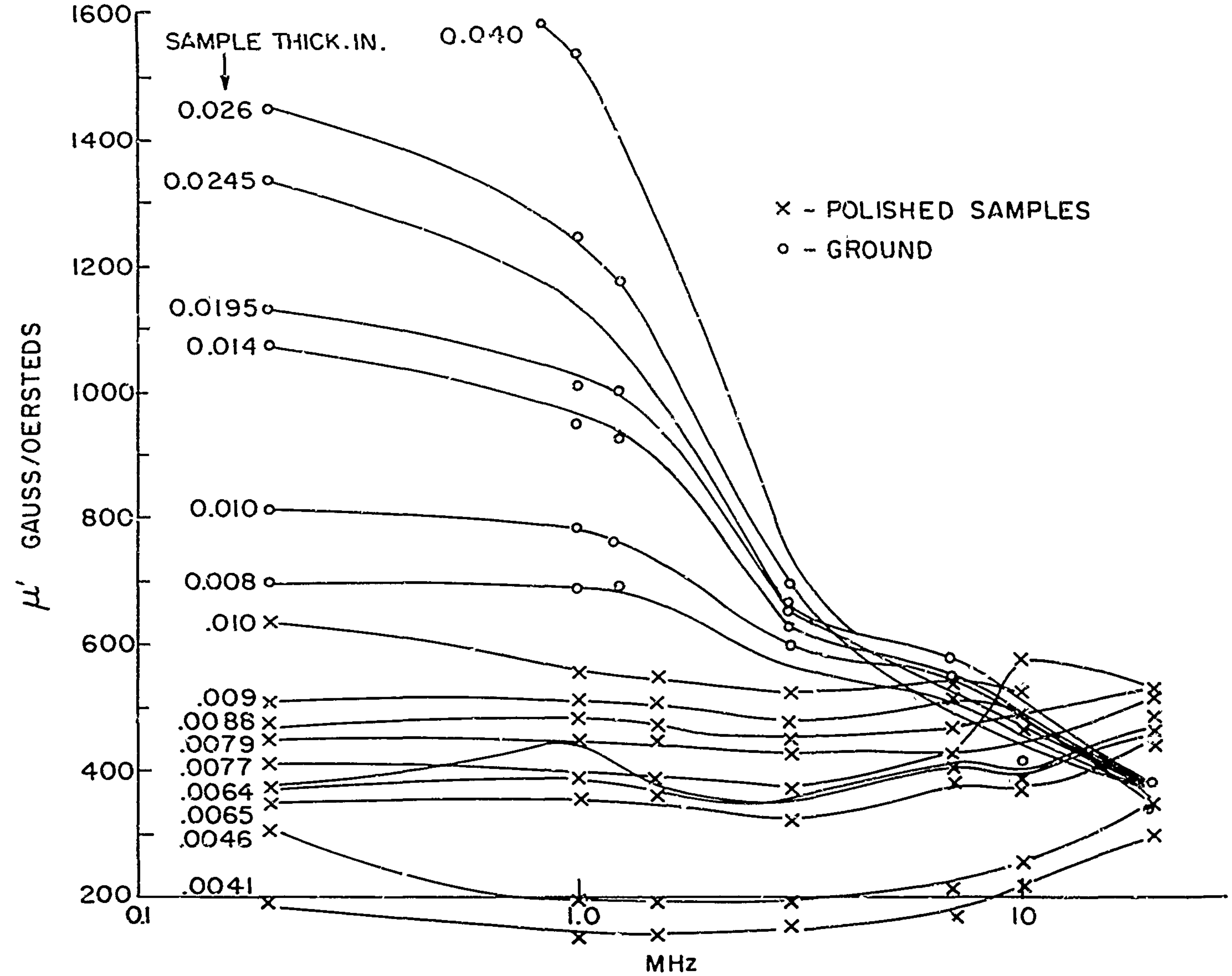

Figure 3. Permeability as a Function of Sample Thickness

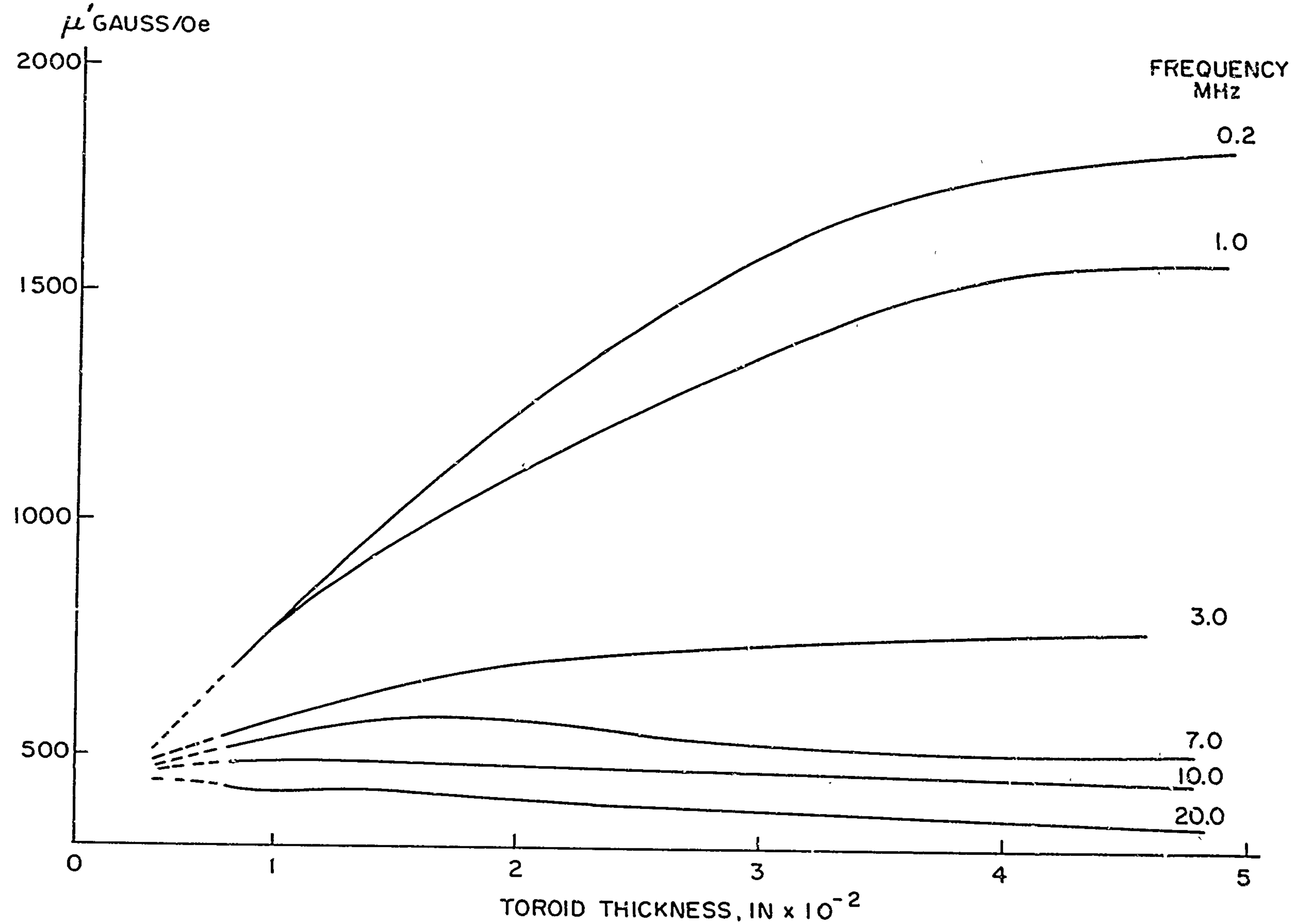

Figure 4. Permeability as a Function of Inverse Sample Thickness

IV. DISCUSSION OF RESULTS

Accepting that the observed variation of magnetic properties in toroids of different thickness is a consequence of different degrees of cold-work in the samples themselves (a point which will be amply confirmed in a subsequent report dealing with effects of annealing), several matters merit discussion:

- The observed structure-sensitivity of magnetic saturation measurements needs to be explained, in view of the widely-accepted notion that B_s is a scalar quantity independent of orientation, grain-size, or stress-state, of a material.

- A reasonable model needs to be found which will account for the dependence of magnetic properties on sample thickness.

- An explanation is needed for the observed differences in behavior of ground and polished samples.

In suggesting a model, we may consider first the observed decrease of saturation magnetization with sample thickness. We are here faced with a three-fold variation in saturation, and the only reasonable explanation seems to be that cold-work must cause a major change in crystal structure of the ferrite, leading to the conversion of significant quantities of the material to a non-ferromagnetic phase.

Some evidence for this explanation is provided by the electron diffraction patterns taken from thin films of the ferrite. From Figure 5 it may be seen that the single-crystal spots are exhibiting splitting, and a hexagonal symmetry appears to be present along with the expected cubic one. This, of course, is typical of face-centered cubic materials containing hexagonal stacking faults due to intense cold-work, and it is possible that the faulted material might be non-ferromagnetic since the environment of the magnetic ions will be changed from that present in the parent lattice.

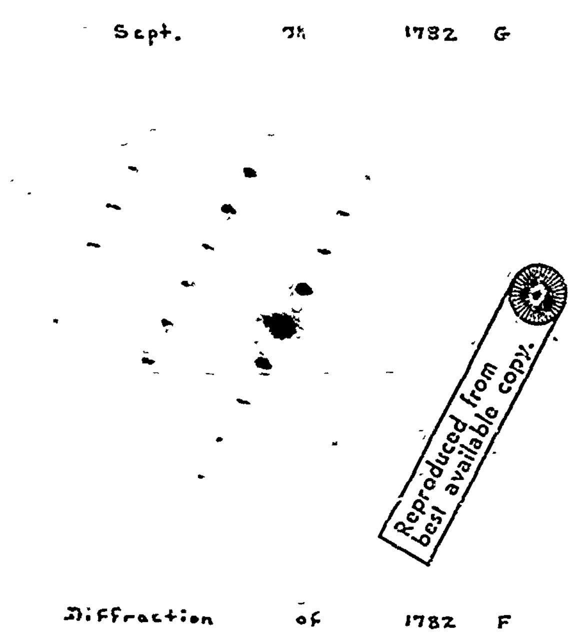

Figure 5. Electron Diffraction Pattern of Cold-Worked Ferrite

A tentative model must therefore allow for the presence of (possibly) non-magnetic inclusions, as well as large internal elastic stresses, in cold-worked ferrite. In the case of saturation magnetization, only the former defects would be expected to be significant, allowing us to propose a simple explanation of the observed variation with sample thickness. Since the variation is non-linear, we may suppose it to be related to the surface/volume ratio, as if the stacking faults were confined to the very skin of the toroids.

Consider the model shown in Figure 6. If the measured value of saturation magnetization, B_A, can be regarded as the arithmetic average of the corresponding saturation values of B_U, in undamaged material, and B_D for damaged, then;

$$B_A \, x = B_D \, 2t + B_U \, (x-2t) \tag{1}$$

$$\text{or} \quad B_A = B_U - \frac{2t}{x} \, (B_U - B_D) \tag{2}$$

B_A and x are the only variables in this case, so a plot of measured saturation magnetization against the reciprocal of the measured sample thickness should yield a straight line of slope $-2t \, (B_N - B_D)$ with an intercept B_U at $1/x = 0$. As may be seen from Figure 7, this seems to hold for polished samples; interestingly enough, ground samples yield a plot which deviates from the straight line at large $1/x$ values. Such a deviation might be expected to occur when $2t = x$, since undamaged material would be left, and equation (2) becomes

$$B_A = B_D = \text{constant} \tag{3}$$

The implication of this difference in behavior between ground and polished samples will be considered later.

Turning to the other magnetic properties, H_C, B_R, and μ', it is to be expected that a more complicated dependence upon sample thickness will be

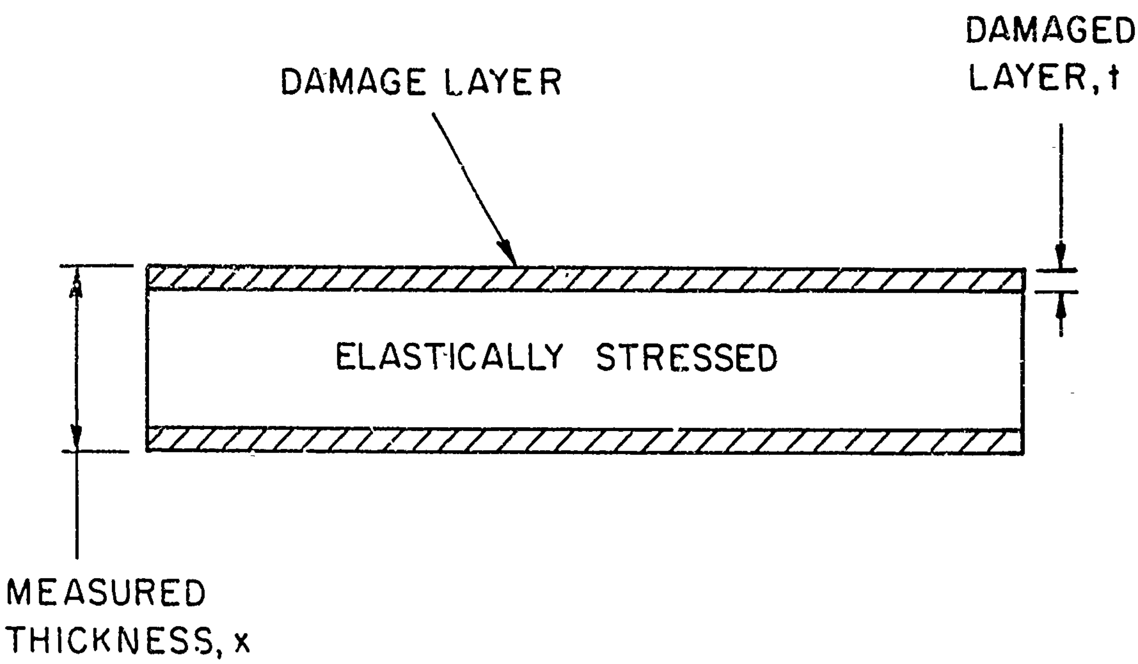

Figure 6. Model of Damage in Ferrite

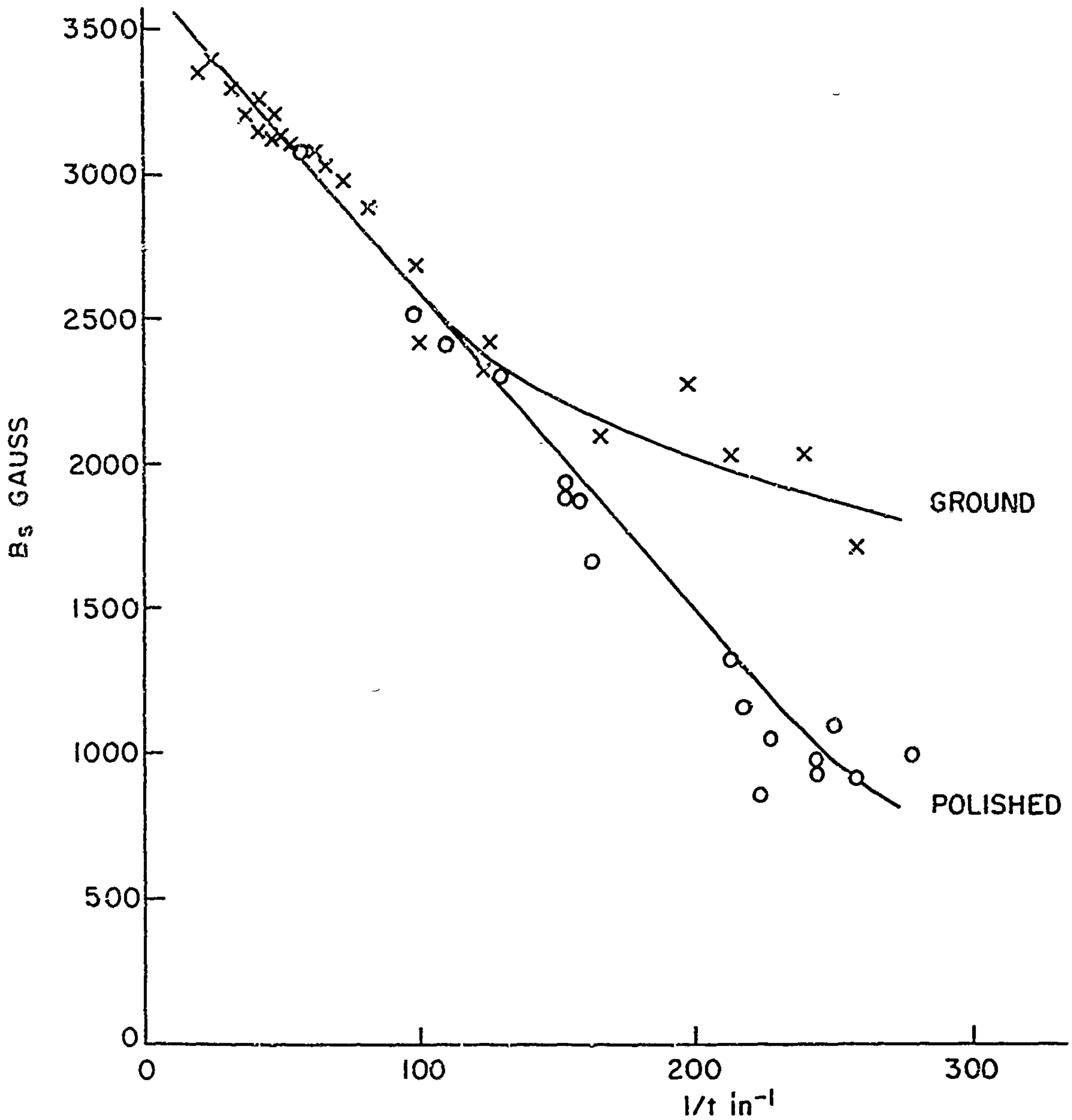

Figure 7. Saturation Magnetization as a Function of Inverse Sample Thickness

evident since, unlike B_S, these properties are sensitive to internal stress. In general terms, however, one might expect B_R to vary with $1/x$ in much the same way as B_S does, since it is usually held that the ratio B_r/B_s in cubic materials has a constant value ~0.6[5].

From Figure 8 it may be seen that a plot of B_r versus $1/x$ does indeed exhibit a straight line region for thicker samples, but levels off at much smaller values of $1/x$ than was the case for saturation magnetization. Once again, the polished samples follow the rule further than do ground ones.

It has frequently been noted that B_S and H_C tend to be inversely related, as in the equation $H_C \propto 1/B_s$ (domain wall energy terms)[6] so one might expect a plot of $1/H_c$ versus $1/x$ to be similar in nature to the preceding graphs. Figure 9 appears to confirm this observation.

Low frequency permeability is notoriously sensitive to stress, but would not be expected to depend on saturation magnetization or the appearance of non-magnetic inclusions. As may be seen from Figure 10, a plot of μ' at 0.2MHz versus $1/x$ shows no straight line portion even though the general tendency of the graph is similar to the others – once again the difference in behavior of polished and ground toroids is marked.

High-frequency permeability, say at 20 MHz, has already been noted to increase as the sample dimension decreases (see Figure 4). This surely reflects the fact that permeability at high frequencies is independent of domain-wall motion or domain rotation, but instead is a dielectric property involving charge oscillations in individual ions [7]. Presumably internal stresses and crystal defects may actually facilitate charge oscillations by weakening the overall regularity of the crystal field.

We may lastly consider the implications of the difference in behavior of polished, and of ground samples. With one noticeable exception, magnetic property changes in thin samples are invariably greater in polished toroids.

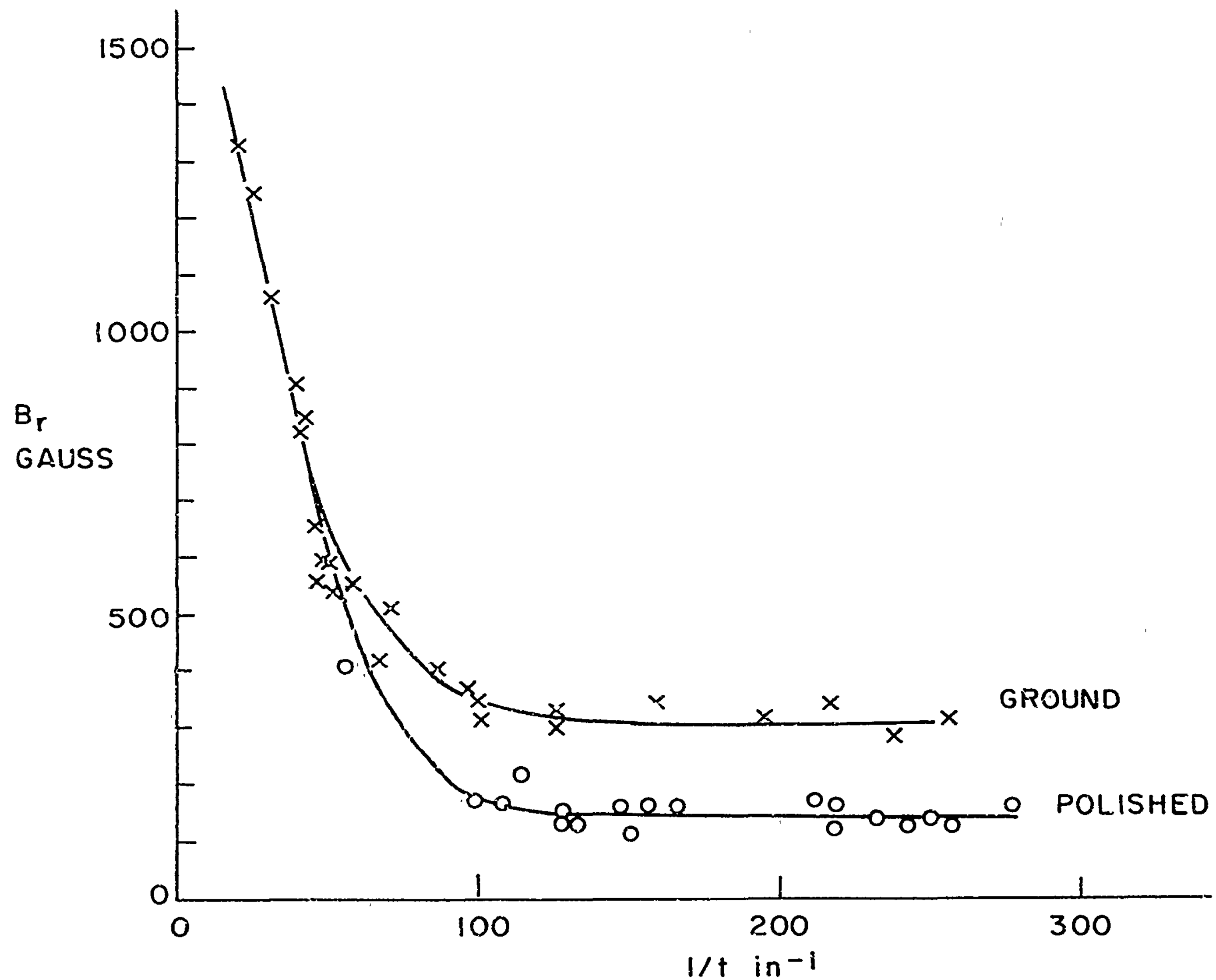

Figure 8. Remanence as a Function of Inverse Sample Thickness

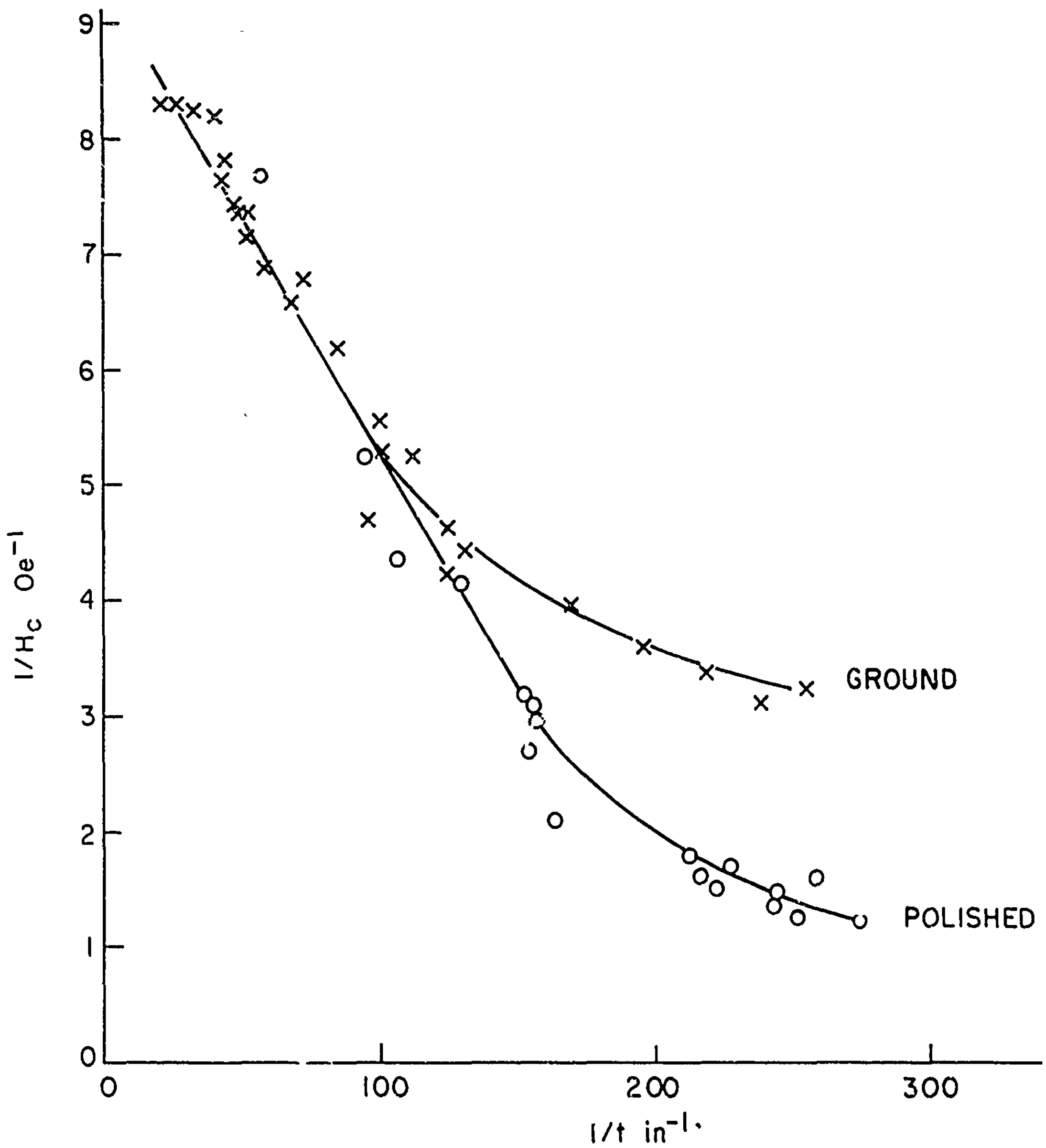

Figure 9. Reciprocal Coercivity as a Function of Inverse Sample Thickness

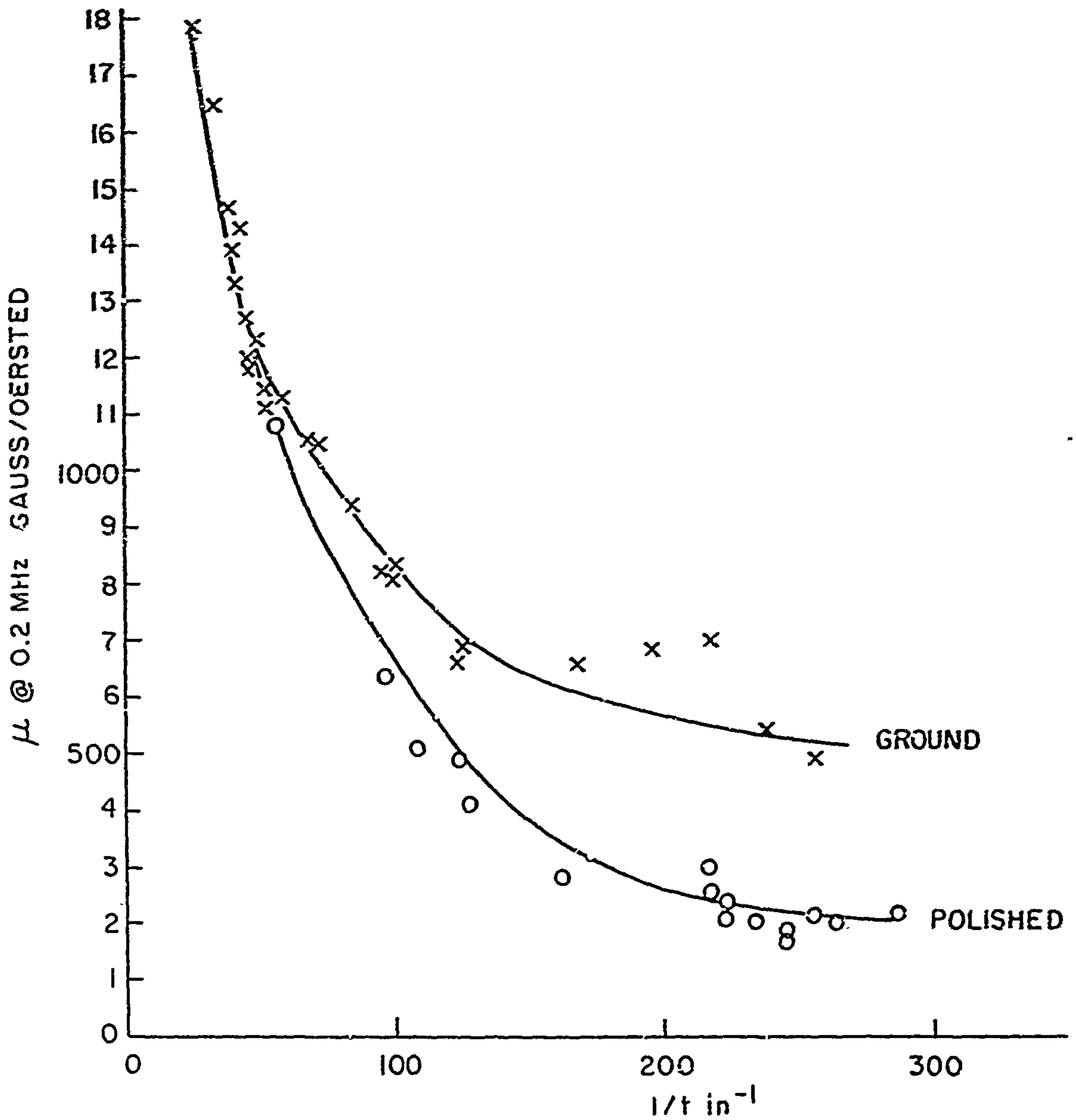

Figure 10. Low Frequency Permeability as a Function of Inverse Sample Thickness

The one exception, as may be appreciated from Figure 4, is the permeability at very high frequencies.

This observation clearly suggests that the damage introduced by polishing is more severe than that produced by grinding; the permeability curves further suggest that the damage is principally experienced as residual stress in all samples. However, the saturation magnetization curves of Figure 7 do imply further that if stacking faults are responsible for B_S changes then they must be produced in a thinner surface layer during polishing than during grinding, since deviation from linearity is slight in the former case. One might infer that the skin produced by polishing is less than 0.002 inch-thick, while that due to grinding is about 0.004 inch.

In addition, it would seem that polishing creates larger internal stresses than grinding, although they are confined to a more concentrated layer. Such an hypothesis fits well with the general experience that rough grinding introduces surface cracks by a fracture mechanism, while polishing causes more plastic flow. Evidently cracking could serve to reduce residual internal stresses which may be locked in by polishing.

V. CONCLUSIONS

The effects of cold-work upon the magnetic properties of nickel-zinc ferrite toroids can be rationalized in terms of residual stresses together with the appearance of a stress-induced phase change.

Polishing and grinding have different effects on the magnetic characteristics of the ferrite; the effects of polishing appear to be more severe than those of grinding, and are furthermore restricted to a thinner surface layer.

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